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NEW SCIENTIFIC RESULTS
of the PhD Thesis

Influence of Supplementary Cementitious Materials on The Properties of High Strength Cement Paste at Elevated Temperatures

(prepared for the internal defense)

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Budapest, 2021

1. Background

Conservation and protection of the environment have become a serious issue in the global world context. Therefore, research on sustainable technologies has been the interest since the early 21st century *(Suhendro, 2014)*. Due to its diverse benefits, concrete is the most common material widely used in the construction industry, producing approximately 20 billion tons of raw materials every year *(Fredonia, 2011)*. Cement production, which is an essential part of concrete production, has primarily been contributing to the increase in the world cement demand to over 3.6 billion tons annually in 2011 *(European Cement Association, 2011)*. The latter amount is expected to be doubled in 2050 *(Scrivener, 2012)*. The industry of cement requires large quantities of natural resources resulting in huge quantities of CO_2 emissions induced over different phases of production of cement *(Malhotra and Mehta, 2005)*. On the other hand, the annual global productions of supplementary cementitious materials (SCM) like metakaolin (MK), silica fume (SF), fly ash (FA) is significantly increased. For instance, amounts of waste FA and slag are approximately 1 billion tons and 360 million tons, respectively *(Reynold, 2009; Juenger and Siddique, 2015)*. Including these large amounts of the waste of SCM as substitutions for cement could be the way of decreasing the global rising of cement demand and its environmental impacts. The constructional richness added to concrete due to the presence of SCM is enormous at both fresh and hardened states. For the fresh properties of concrete, SCM are used to increase the workability of concrete and simplifies the implementation process of complex design structural elements *(El Mir and Nehme, 2017)*. From another hand, different mechanical properties are improved on the hardened concrete such as increased toughness, improved the bond strength, and improved the durability due to the very low permeability *(Sabir et al., 2001; Siddique, 2011; Merida and Kharchi, 2015; Alcamand et al., 2018)*. The chemical compositions, degree of crystallinity, and fineness of SCM are the factors for the improvement of the hardened property of concrete as well. Furthermore, the pozzolanic reaction of SCM with the $Ca(OH)_2$ resulted from the hydration product of cement produces additional strength. Besides the provided strength by these materials, SCM are used for filling the skeleton of concrete, thus enriching macro/micro-structure, and resulting in reducing the permeability and producing high dense concrete *(Sata et al., 2007)*. The advantages of SCM are restricted by the occurrence of cracking and in some cases spalling, particularly in the case of SF that happens due to two main factors correspond to heat pressure and high dense microstructure provided by SCM *(Hertz, 1992)*. However, recent fire cases and laboratory studies showed disastrous deterioration of high strength concrete (HSC) after exposure to elevated temperatures *(Ma et al., 2015)*. Since fire is an unexpected incident and has an impact on the properties of concrete, therefore, identify, evaluate, and enhance the behaviour of HSC at elevated temperatures is of great importance *(Shah et al., 2019; Fehérvári and Nehme, 2009)*. The endeavour of blending cement with replacements of SCM to produce heat-resistance concrete is a promising attempt in the field of concrete subjected to elevated temperatures.

2. Research Significance and Objectives

The concept of adoption of waste materials in construction is a quantum leap for not merely cleaning the environment but also introducing them into construction, thereby further contributing to improving the properties of concrete. Therefore, the sustainability of engineering products is not a choice anymore but a demand. HSC based on SCM is a new generation of concrete that is based on the concepts of high-performance concrete (HPC) and fire-resistant concrete. An eco-friendly version of this concrete can be developed by the replacement of specific amounts of cement with amounts of SCM, where sustainable and economic value could be added. The significance of incorporating SCM into concrete mixtures to produce HSC with enhanced properties against fire is of great importance for the benefit of the environment, economy, and different engineering applications.

The behaviour of HSC based on SCM at elevated temperature has shown different thermal and mechanical properties compared to the ordinary HSC. To evaluate the fire resistance of HSC based on SCM, there is a need to characterize the physical, chemical, and mechanical properties of the constituents including the SCM. One of the major properties that are affected in HSC is related to spalling. To obtain beneficial effects of SCM regarding spalling as well as stabilizing the hardened cement paste from different harmful chemical composition, comprehensive experimental investigations have been carried out by testing several parameters, i.e., different types and amounts of SCM, different ratios of water to binder (w/b), different types of cement fineness, and different levels of elevated temperature. Therefore, the main objective of the study is to investigate the properties of the high strength cement paste (HSCP) using different types and amounts of SCM at elevated temperatures. In order to fulfil the main objective, the current study aims to address the following sub-objectives:

- Defining the optimum replacement of using MK in HSCP after exposure to elevated temperatures.
- Defining the optimum replacement of using SF in HSCP after exposure to elevated temperatures.
- Defining the optimum replacement of using FA in HSCP after exposure to elevated temperatures.
- Determining the effect of changing w/b ratio on the optimum replacements of using SCM in HSCP after exposure to elevated temperatures.
- Determining the effect of changing the fineness of cement on the optimum replacements of using SCM in cement paste after exposure to elevated temperatures.

Several laboratory experiments were conducted on the produced HSCP. In addition, different properties that were directly interconnected with the macro and micro-structure of the pastes were also investigated using several tests and techniques.

3. Experimental Program

Several HSCP mixtures have been produced depending on different variables. The variables corresponding to the HSCP mixtures are (i) three w/b ratios, i.e., 0.30, 0.35, and 0.40, (ii) different types of SCM, i.e., MK, SF, and FA, (iii) different replacement amounts of SCM, i.e., 0%, 3%, 6%, 9%, 12% and 15% by cement mass, (iv) two types of cement fineness, i.e., CEM I 42.5 N and CEM I 52.5 N, and (v) different levels of temperature, i.e., 20, 50, 150, 300, 400, 500, 800, and 900 °C.

A total of 66 mixtures of HSCP, including approximately 1600 cubes and 1600 prisms, were prepared and experimentally tested. Three samples were considered for each mixture. The prepared mixtures are divided into two groups, as shown in **Figure 1**. The first group is prepared by CEM I 42.5 N (the right branch of **Figure 1**), whereas mixtures in the second group were made with CEM I 52.5 N (the left branch of **Figure 1**).

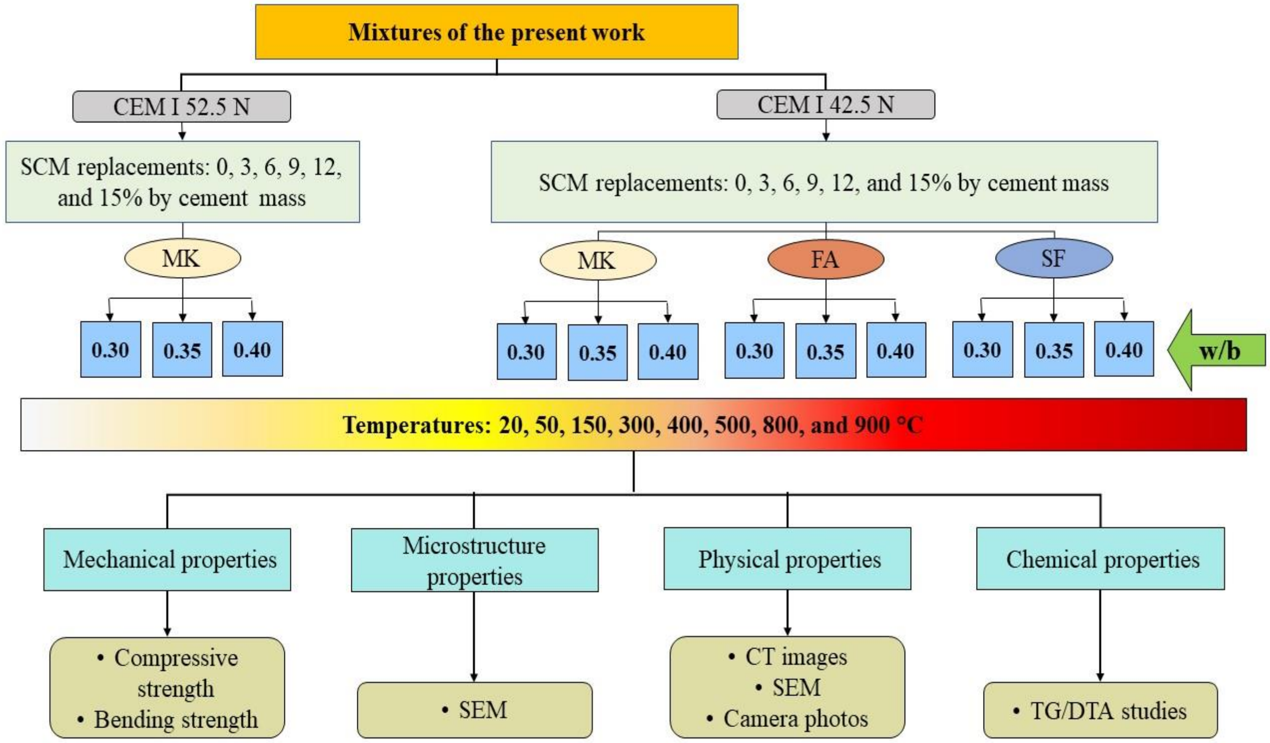


__Figure 1__ Parameters of the mixtures for the experimental work

3.1 Heating and testing procedures

The procedures of heating and testing have been prepared as follows:

1. Mixing and casting cement paste to produce cubes and prisms.
2. Around 24 hours after the casting, the cubes were de-moulded then placed in the water tank.
3. After 7 days of curing, the cubes and prisms were removed from the water tank then stored in the lab conditions (20 ± 2 °C and 35% relative humidity).

4. At the age of 90 days, the testing program of specimens including heating was conducted as recommended by RILEM *(RILEM Technical Committees 129-MHT, 1995)*.
5. The heating program was applied by heating specimens to the target temperature (20, 50, 150, 300, 400, 500, 800, and 900 °C,) for a duration of two hours of exposure.
6. Finally, after the heating process, the cubes were left in the lab condition for 24 hours to be cooled down, then the load-bearing tests were applied for the cold state.

3.2 Mechanical properties

The tested mechanical properties are compressive strength and three-point bending strength tests. Tests follow the Standards *MSZ EN 12390-3 (2009)* for the compression test, and *MSZ EN 12390-5 (2009)* for the bending test. The tests were comprehensively carried out to investigate the mechanical properties of the specimen at ambient and different levels of maximum temperatures.

3.2.1 Compressive strength

Specimens (cubes) with a size of 30×30×30 mm were used for hardened cement paste investigations in compression. The test machine was ALPHA 3/3000S at a constant loading rate of 1.40 kN/s. For concrete, cubes of 150x150x150 mm were tested by a universal closed-loop hydraulic machine with a constant rate of 11.25 kN/s.

3.2.2 Bending strength

Specimens with a size of 40×40×160 mm prisms were tested by conducting a three-point bending strength test. The test machine was ALPHA 3/3000S at a constant loading rate of 0.06 kN/s. Whereas for concrete, prisms of 70×70×250 mm were tested by a three-point bending test as well.

3.3 Thermogravimetric program

TG investigation defines the ranges of various thermal decompositions of different hardened paste products and phases with simultaneous estimation of the mass loss in static condition. Moreover, changes in phases are controlled by TG/DTG/DTA, using MOM Derivatograph-Q 1500 D TG/DTA instrument. The tested powders were taken from specimens of ambient temperature 20 °C and selected from the core of specimens. The investigated powders are taken from the samples that showed high performance after exposure to elevated temperatures (optimum replacements of SCM), as well as the mixtures of pure OPC (reference) for comparison.

3.4 Computed Tomography (CT) study

The examination of permeability, pores, and pore size distribution of hardened cement paste can be obtained by using the CT method *(Lublóy et al., 2015; Balázs et al., 2017)*. The CT tests were carried out by a Siemens Somatom 16 device at the Diagnostic and Oncoradiology Institute of Kaposvar University in Hungary. The thicknesses of slices were 1.5 mm and the pixel spacing was 0.225 mm.

CT measurements have been carried on cylindrical specimens with a diameter of 100 mm and a height of 50 mm for both the reference mixture and the mixture with SCM.

3.5 Scanning electron microscope (SEM)

Scanning electron microscope investigations have helped to discover and understand the microstructure of hardened cement paste as well as the interfacial transition zone (ITZ) of concrete. SEM offers greater magnification and resolution to the microstructural level of the modified HSCP *(Tam et al., 2020)*. The tested samples were prepared with a smooth viewing face in order to obtain a better image resolution. SEM images were taken for both reference mixtures and mixtures containing SCM from the core of the cross-sectional areas of the specimen. Then the samples faces were coated with golden spray for 30 s in a vacuum chamber, and thereafter, the samples are investigated using Phenom XL SEM in the lab of Budapest University.

3.6 High strength concrete mixtures

In order to understand the connection between the hardened cement paste and concrete mixtures, additional concrete mixtures have been prepared and tested after elevated temperatures. The ITZ of aggregate and hardened cement paste in the presence of SCM is provided herein as well. The proportions of the aggregate fractions are 45% for sand with a size of 0-4 mm, 30% for small gravel with a size of 4-8 mm, and 25% for medium gravel with a size of 8-16 mm.

4. New Scientific Results (NSR)

Several parameters have been investigated on the residual mechanical properties of HSCP after exposure to elevated temperature. These parameters include different types and ratios of SCM, cement fineness, level of elevated temperature, and different w/b ratios. All tests are carried out at the age of 90 days in a cold state in terms of compressive and bending strength tests. The relative residual compressive strength (RRCS) is expressed by the division of the residual strength for each level of temperature to the residual strength of the same mixture at 20 °C. The following NSR are related to the main objectives of the study.

NSR 1: Influence of supplementary cementitious materials on the mechanical properties of high strength cement paste after exposure to elevated temperatures

A total of 48 mixtures of HSCP made of CEM I 42.5 N have been investigated in the current study. Results of the following NSR are corresponded to compressive strength tests for three types of SCM, i.e., MK, SF, and FA. Since the factor of w/b ratio has a separate section in the NSR (NSR 2.3), only the results of w/b of 0.30 have been considered herein.

The optimum replacement of SCM is considered to be the highest value of the relative residual strength at temperatures of 500 and 800 °C. In addition, the heat resistance method (area under the curve) is used to support the results of the optimum replacements.

1.1: I experimentally proved that incorporating metakaolin material as a partial replacement of cement mass enhances the mechanical properties of cement paste after exposure to elevated temperatures. As a result, 9% of metakaolin replacement to cement has been considered to be the optimum value, increasing the strength by about 52% and 39%, compared to the reference samples exposed to the same temperatures, in terms of relative residual compressive strength after exposure to 500 and 800 °C, respectively [1, 2, 4, 6, 17].

Figures 2 and **3** show the results of the compressive strength tests and the measured heat resistance. Results are supported by physical and chemical investigations. The physical observations have shown that the presence of MK decreases the size and number of cracks keeping the body of HSCP coherent compared to the reference mixture that showed several disintegrations. The chemical investigations have been conducted by means of TG/DTA and SEM on the optimum replacement of MK (9%) and the reference mixture (0%). Results showed that presence of MK decreases the harmful effect of $Ca(OH)_2$ decomposition after exposure to elevated temperature. The replacements amount of MK above the optimum replacement (12% and 15%) showed less RRCS compared to the optimum replacement after exposure to elevated temperature. This could be attributed to the high dense structure provided at high MK replacements which induce internal cracking.

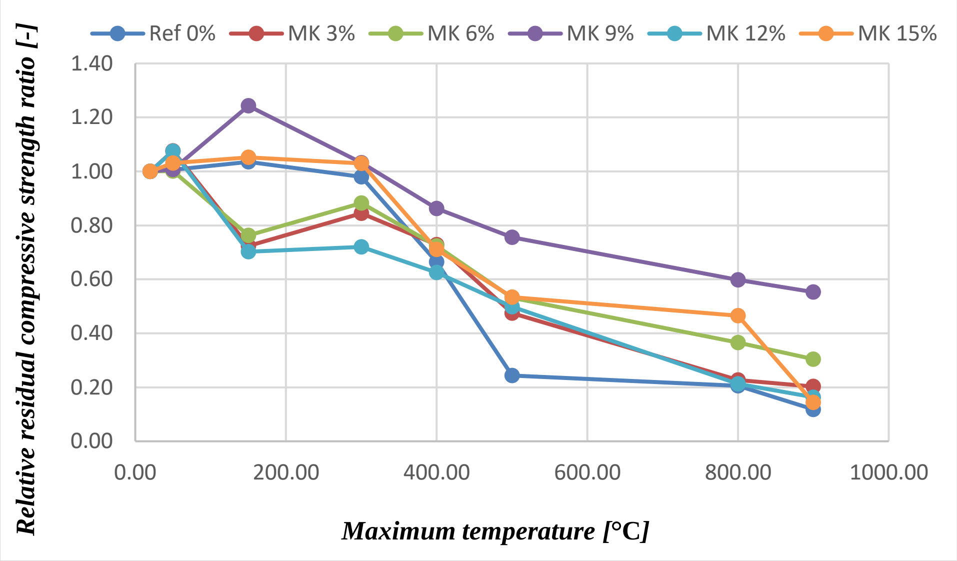


***Figure 2** Relative residual compressive strength for different metakaolin replacements with 0.30 w/b ratio at different temperatures exposures*

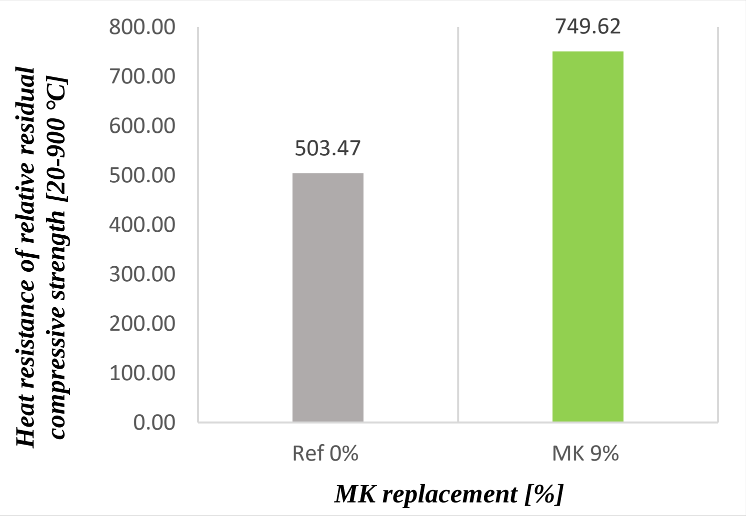


***Figure 3** Heat resistance results of reference mixture and mixture containing 9% of metakaolin (MK 9%; 20–900 °C)*

1.2: I experimentally proved that incorporating silica fume material as a partial replacement of cement mass enhances the mechanical properties of cement paste after exposure to elevated temperatures. As a result, 6% of silica fume replacement to cement has been considered to be the optimum value, increasing the strength by about 73% and 43%, compared to the reference samples exposed to the same temperature, in terms of relative residual compressive strength after exposure to 500 and 800 °C, respectively [1, 13, 15, 17].

Figures 4 and **5** show the results of the compressive strength tests and the measured heat resistance. Results are supported by physical and chemical investigations. The physical properties have been conducted using CT measurements on the optimum and reference mixtures. Results have shown that the presence of SF decreases the porosity compared to the reference mixture, which showed several disintegrations after exposure to elevated temperatures. The chemical investigations conducted by TG/DTA and SEM have shown that the presence of SF decreases the harmful effect of $Ca(OH)_2$ decomposition after exposure to elevated temperature.

From another hand, results showed that the highest value of the RRCS at temperature 500 °C is related to 6% replacement of SF whereas at temperature 800 °C, 9% replacement of SF has been observed to be slightly higher than 6% replacement. However, by measuring the heat resistance for all mixtures, 6% replacement of SF has been found to be the optimum value.

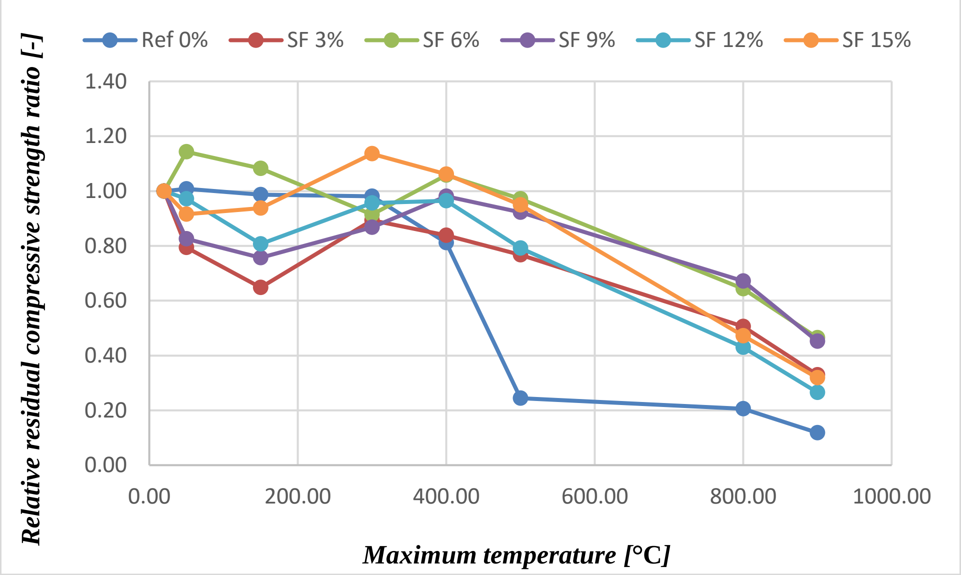


Figure 4 *Relative residual compressive strength for different silica fume replacements with 0.30 w/b ratio at different temperatures exposures*

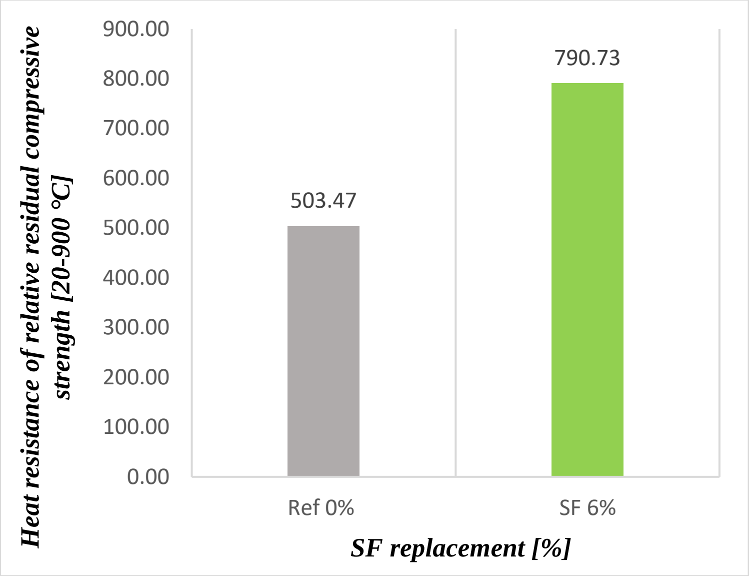


***Figure 5** Heat resistance results of reference mixture and mixture containing 6% of silica fume (SF 6%, 20–900 °C)*

1.3: I experimentally proved that incorporating fly ash material as a partial replacement of cement mass enhances the mechanical properties of cement paste after exposure to elevated temperatures. As a result, 15% of fly ash replacement to cement has been considered to be the optimum value, increasing the strength by about 71% and 36%, compared to the reference samples exposed to the same temperature, in terms of relative residual compressive strength after exposure to 500 and 800 °C, respectively [1, 17].

Figures 6 and **7** show the results of the compressive strength tests and the measured heat resistance. Results are supported by physical and chemical investigations. The physical properties have been conducted using CT measurements on the optimum and reference mixtures. Results have shown that the presence of FA decreases the porosity compared to the reference mixture, which showed several disintegrations after exposure to elevated temperatures. The chemical investigations conducted by TG/DTA and SEM have shown that the presence of FA decreases the harmful effect of $Ca(OH)_2$ decomposition after exposure to elevated temperature. Results showed that the highest values of the RRCS and heat resistance for all mixtures are related to 15% replacement of FA.

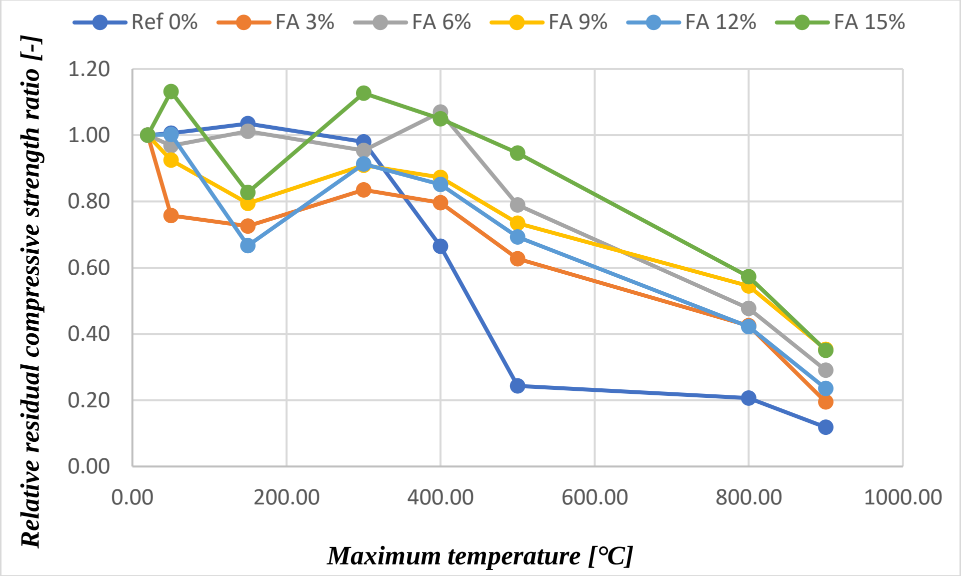


Figure 6 *Relative residual compressive strength as a function of temperature of fly ash mixtures of 0.30 w/b ratio*

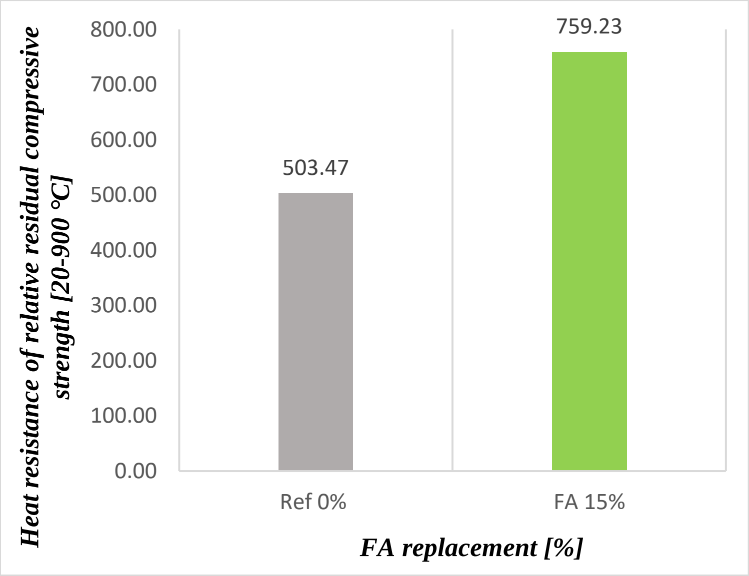


Figure 7 *Heat resistance results of reference mixture and the mixture containing 15% of fly ash (FA 15%, 20–900 °C)*

1.4: I experimentally proved that elevating temperatures up to 800 °C has changed the optimum replacements of supplementary cementitious materials compared to the optimum replacements at ambient temperatures [2, 15].

The amounts of optimum replacements of the three SCM after exposure to elevated temperatures are higher than amounts of optimum replacements at ambient temperatures. The difference between the optimum replacements of ambient and elevated temperatures could be attributed to an important reason; at elevated temperatures the required amounts of SCM to decrease the amount of $Ca(OH)_2$ are higher than amounts of SCM needed at ambient temperature (**Figure 8**). All values confirmed the previous observation except the group of MK with 0.30 w/b ratio.

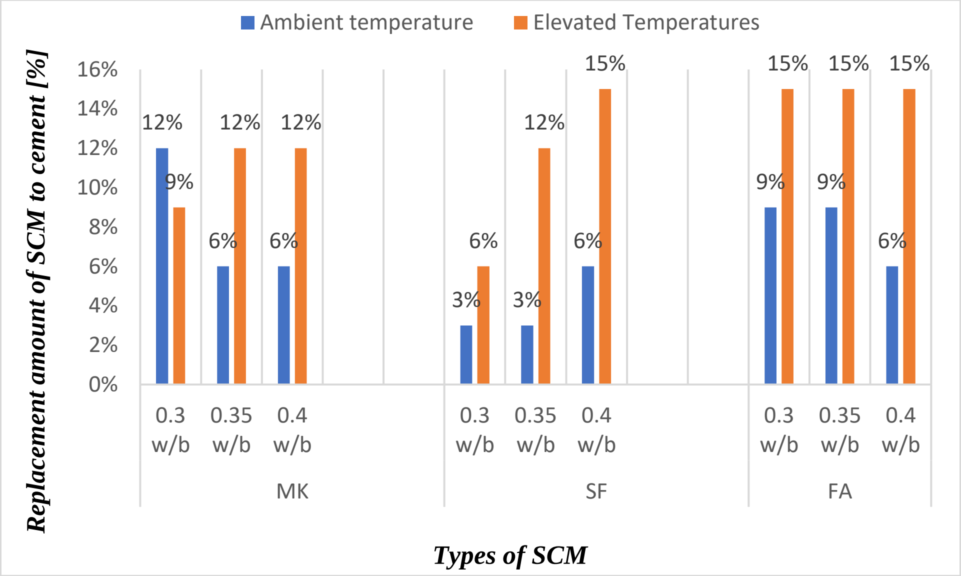


Figure 8 *The optimum replacement values of the used supplementary cementitious materials (by cement replacement) at ambient and after exposure to elevated temperatures*

NSR 2: Parameters affecting the behaviour of supplementary cementitious materials on the high strength cement paste after exposure to elevated temperatures.

Two parameters were considered regarding the behaviour of SCM during the tests; cement fineness represented by testing two grades of cement (CEM I 42.5 N and CEM I 52.5 N), and three ratios of w/b (0.30, 0.35 and 0.40).

2.1: By studying two different grading fineness of ordinary Portland cement (CEM I 42.5 N and CEM I 52.5 N), I have demonstrated that the value of the optimum replacement of metakaolin after exposure to elevated temperatures decreases as the cement fineness increases. This result is valid at w/b ratios 0.30 and 0.35 as long as compressive strength is concerned [1, 2, 8, 9, 14].

The RRCS has shown that the increase in the fineness of cement decreases the amount of MK needed for the optimum value. This observation is confirmed for both 0.30 and 0.35 of the w/b ratios. Using MK as a fine material blended with fine cement (CEM I 52.5 N) have limitations due to the vulnerability to the disintegration of the mix induced by the high densification. This result is more pronounced at low w/b ratio and high elevated temperatures yet, insignificant at a relatively high w/b ratio (0.40), as shown in **Figure 9**.

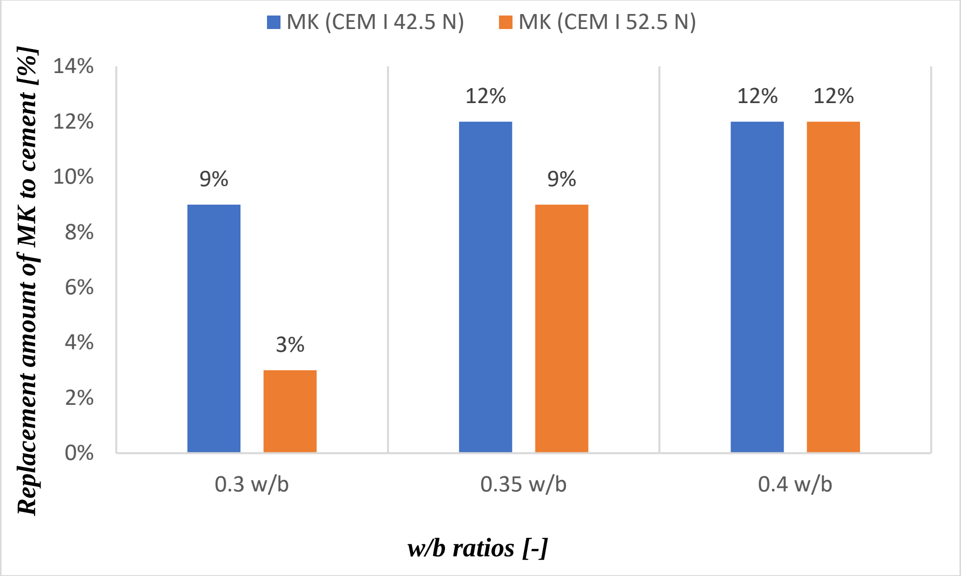


***Figure 9** The optimum replacements of metakaolin with CEM I 42.5 N and CEM I 52.5 N in a function of w/b ratio*

2.2: By studying two different grading fineness of ordinary Portland cement (CEM I 42.5 N and CEM I 52.5 N) on reference mixtures, I have demonstrated that the value of the heat resistance of high strength cement paste after exposure to elevated temperatures (up to 900 °C) increases as the cement fineness increases [1, 2, 14].

Figure 10 shows the results of the heat resistance of the reference pastes at three w/b ratios.

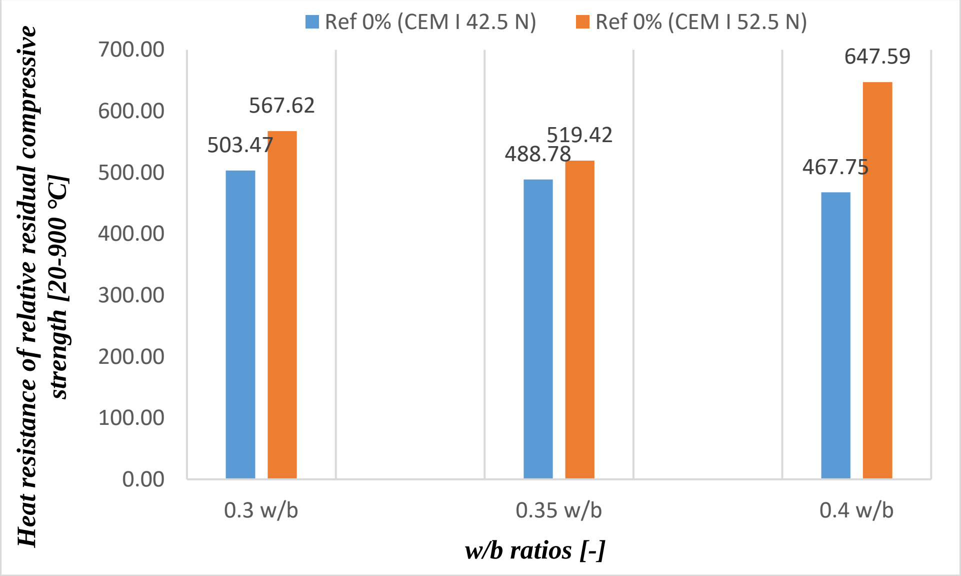


***Figure 10** Heat resistance results of reference mixtures with CEM I 42.5 N and CEM I 52.5 N [20–900 °C]*

2.3: By studying three different w/b ratios (0.30, 0.35, and 0.40) on the compressive strength of the high strength cement paste incorporated with supplementary cementitious materials, I have demonstrated that the value of the optimum replacement amount of supplementary cementitious materials after exposure to elevated temperatures increases as the w/b ratio increases [2, 13, 14, 15].

I have experimentally investigated the effect of changing w/b ratio on the compressive strength of HSCP using CEM I 42.5 N incorporated with MK or SF. In addition, the effect of changing w/b ratio is also investigated on the compressive strength of HSCP using CEM I 52.5 N incorporated with MK only. Results, shown in **Figure 11,** showed that the value of the optimum replacement amount of MK or SF increases as the w/b ratio increases. This result has been justified by the high porosity of the mixes due to the high w/b ratio, in which the higher the porosity of the mixture is the higher the value of the fine material is required.

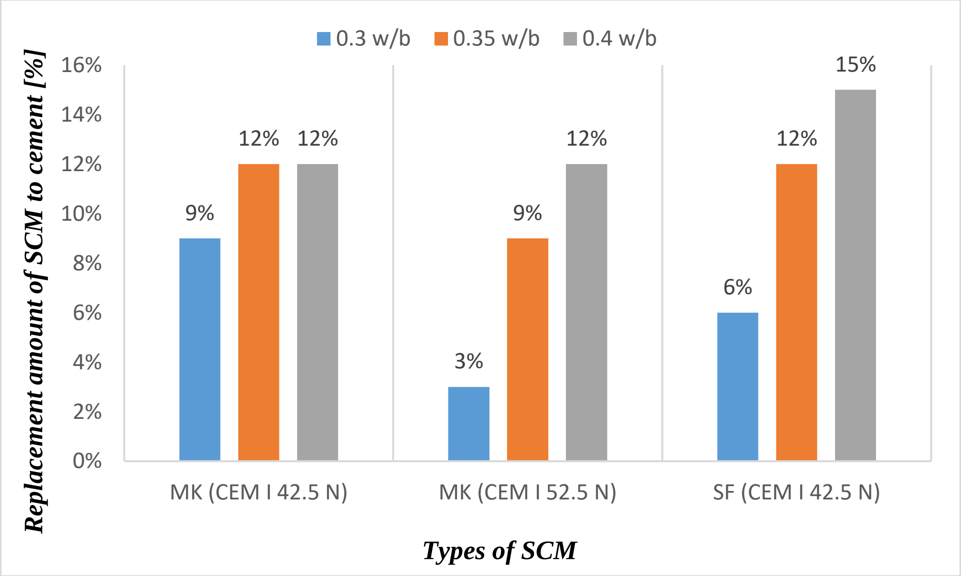


Figure 11 *The optimum replacements of metakaolin and silica fume in the function of w/b ratio*

NSR 3: A comparison between the behaviour of high strength cement paste and high strength concrete after exposure to elevated temperatures.

The finding in this section provides a link between the results obtained from the studies of hardened cement paste and the results obtained from concrete. The relationship is obtained by applying the optimum replacement of MK in hardened cement paste to produce high strength concrete. Production of heat-resistant hardened cement paste is of a great chance to enhance the heat-resistance of HSC.

3.1 By applying the obtained optimum replacement of metakaolin of cement paste (9%) in concrete mixture, I have experimentally proved that the values of compressive strength of concrete are in a good agreement with the values of compressive strength of hardened cement

paste. This result is limited to one type of cement (CEM I 52.5 N), one w/b (0.35), and one type of aggregate (normal river aggregate) [18].

Cement paste has unstable conditions in concrete during heating due to several chemical and physical changes, particularly from 60 °C to 800 °C *(Khoury, 1995)*. Thus, the heat-resistance hardened cement pastes have chances to significantly enhance the heat-resistance properties of concrete after exposure to elevated temperature. From our results, for mixtures of 0% MK (reference mixtures), the ratios of concrete mixtures (HSC) to the cement paste mixtures (HSCP) are 1.08 and 2.69 at temperatures 20 and 800 °C, respectively. For mixtures containing optimum MK replacement (9% of MK), the ratios of concrete to cement paste are 1.15 and 0.96 at temperatures 20 and 800 °C, respectively. This indicates the compatibility between concrete and cement paste obtained by the presence of MK at elevated temperatures. Results are also illustrated in **Figure 12**.

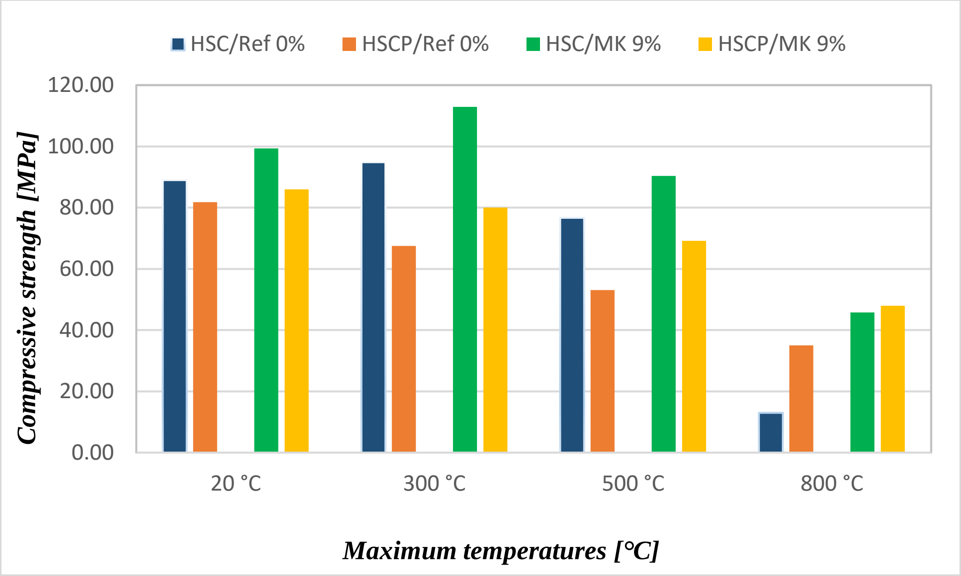
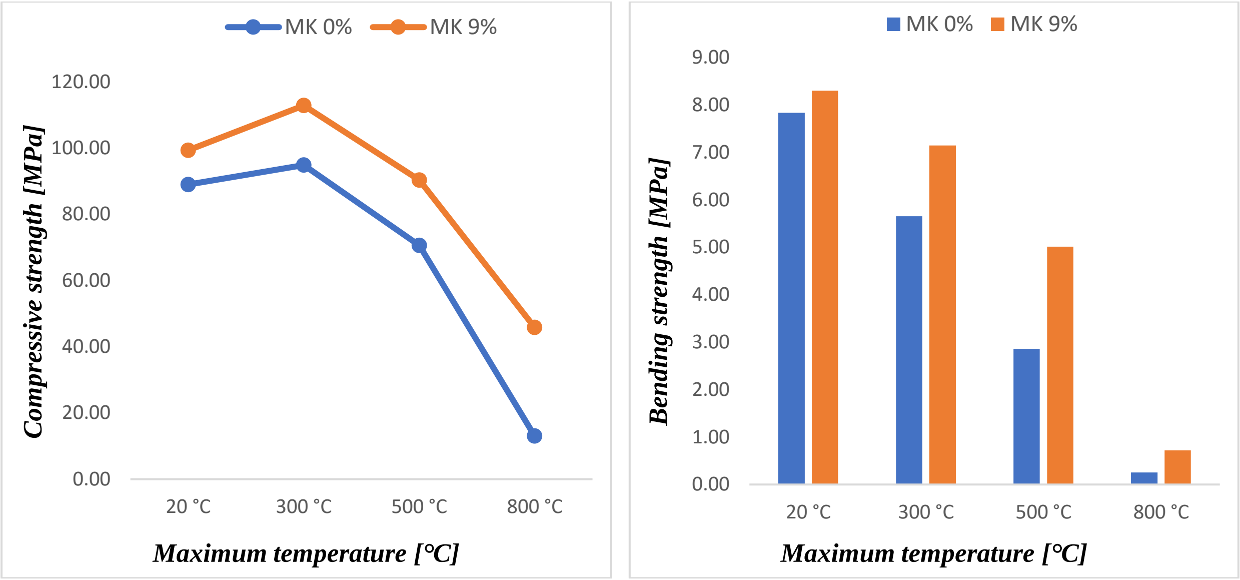


Figure 12 *Variation of high strength concrete compressive strength values with respect to metakaolin replacements and temperature levels*

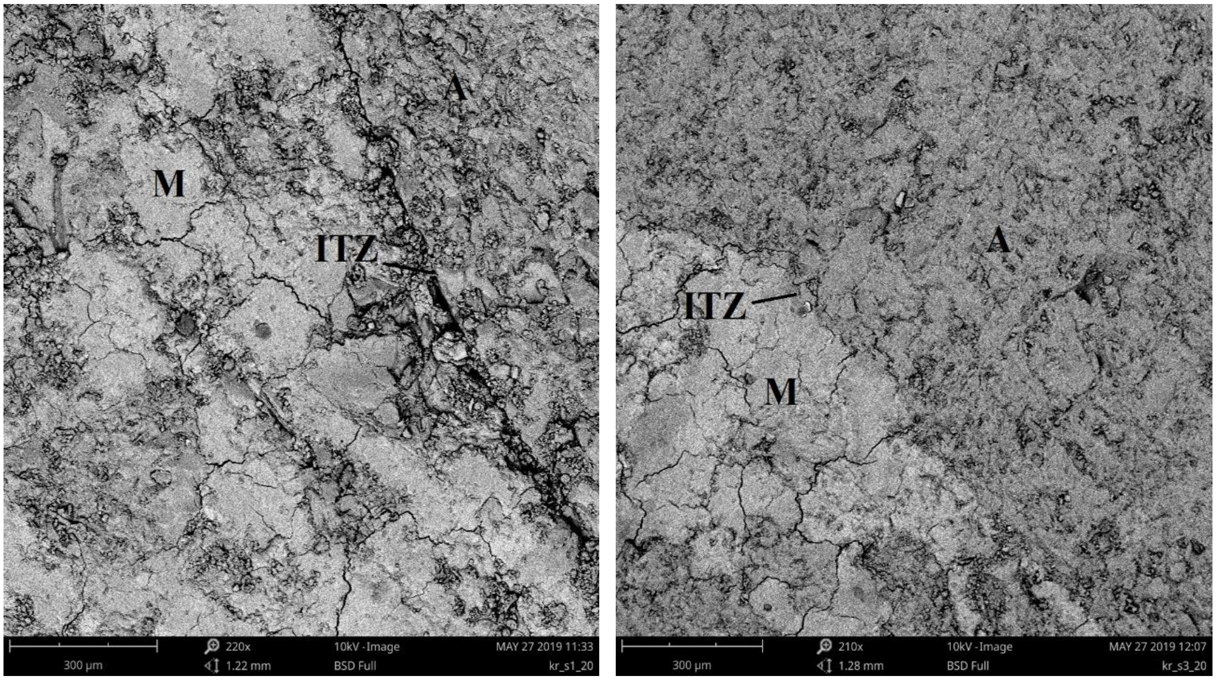
3.2: By using the scanning electron microscope, I proved that the presence of metakaolin significantly enhances the interfacial transition zone between mortar and aggregate for the high strength concrete. This result is limited to one type of cement (CEM I 52.5 N), one w/b (0.35), and one type of aggregate (normal river aggregate) [18].

The results are obtained by testing two types of concrete mixes (reference mixture and mixture containing 9% of MK). Using MK enhances the compressive and bending strength results in all levels of temperatures **Figures 13**. Besides, the conducted tests for the mechanical properties are supported by SEM images, as shown in **Figures 14**. Images clearly showed that the presence of MK has mitigated the borderline between the mortar and the aggregate, enhancing the ITZ. This could be as a result of the filler effect and pozzolanic reaction provided by MK.



a) b)

Figure 13 *Variation of high strength concrete mechanical properties strength with respect to metakaolin replacements and temperature levels: a) compressive strength, b) bending strength*



HSC/MK (0%) HSC/MK (9%)

Figure 14 *The interfacial transition zone between the mortar of high strength concrete and the aggregate at the reference concrete (HSC/MK (0%)) and concrete with MK (HSC/MK (9%))*

5. List of publications

1. **Abdelmelek, N**. and Lubloy, E. 2021. "The impact of metakaolin, silica fume and fly ash on the temperature resistance of high strength cement paste". Journal of Thermal Analysis and Calorimetry, pp.1-12.
2. **Abdelmelek, N**. and Lubloy, E., 2020. "Evaluation of the mechanical properties of high-strength cement paste at elevated temperatures using metakaolin". Journal of Thermal Analysis and Calorimetry, pp.1-15.
3. Balázs, G.L., Kopecskó, K., Alimrani, N., **Abdelmelek, N**. and Lublóy, É., 2018. "Fire Resistance of Concretes with Blended Cements". In High Tech Concrete: Where Technology and Engineering Meet (pp. 1420-1427). Springer, Cham.
4. **Abdelmelek, N**. and Lublóy, É., 2017. "Improved fire resistance by using different dosages of metakaolin". 12 th Central European Congress on Concrete Engineering. 31 August to 1st September 2017. 675p. Conference location, time: Tokaj, Hungary, FIB Magyar Tagozata, 2017. pp. 240-246. http://fib.bme.hu/konyvek/ccc2017.pdf
5. Balázs, G.L., Kopecskó, K., Alimrani, N., **Abdelmelek, N**., Lublóy, É. 2017. "Fire Resistance of Concretes with Blended Cements". Proceedings, "High Tech Concrete: Where Technology and Engineering Meet" (Eds. Hordijk, D.A., Lukovic, M.), fib Symposium 12-14 June 2017 Maastricht, The Netherlands, Springer, pp. 1420-1427. www.springer.com
6. **Abdelmelek, N**., Lublóy, É. 2018. "Effect of Metakaolin on Mechanical Properties Of Cement Paste Exposed To Elevated Temperatures". Proceeding of the 12th fib International PhD Symposium. PP 3-11, 2018, Czech Technical University in Prague, Prague, Czech Republic. http://phdsymp2018.eu/dwn/Proceedings
7. Éva, Lublóy., György, Baláz.s, Salem, G. Nehme., Katalin, Kopecskó., **Nabil Abdelmelek**., Viktor, Hlavicka. 2018. "A Betonfelület Leválásának Hatása A Szerkezet Tűzállóságára". EPKO 2018, 22nd International Conference on Civil Engineering and Architecture. pp 141-146.
8. **Abdelmelek, N**. and Lublóy, É. 2019. "Evaluating compressive strength of cement paste at elevated temperatures using metakaolin". Third international fire safety symposium 2019, in 5-7 June, Canada, PP 311-319.
9. **Abdelmelek, N**. and Lublóy, É. 2019. "Behaviour of high strength paste prepared with metakaolin at elevated temperature". *fib* Symposium 2019, May 27-29, Krakow, Poland, PP. 663-665.
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